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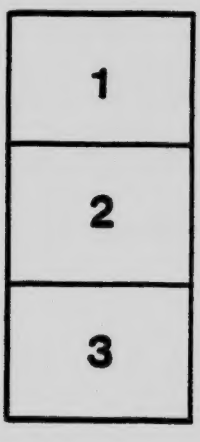

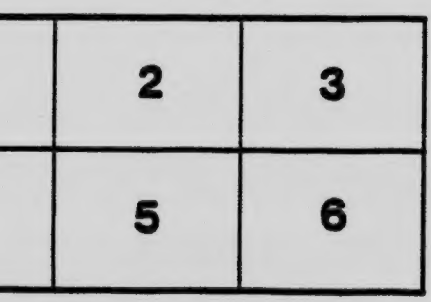

DEPARTMENT OF AGRICULTURE
BRANCH OF THE DAIRY AND COLD STORAGE COMMISSIONER
OTTAWA, CANADA

APPARATUS FOR THE DETERMINATION OF WATER AND FAT IN BUTTER

BY
FRANK T. SHUTT, M.A.
Chemist, Dominion Experimental Farms

BULLETIN No. 14
Dairy and Cold Storage Commissioner's Series

Published by direction of the Hon. SYDNEY A. FISHER, Minister of Agriculture, Ottawa, Ont.

February, 1907

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LETTER OF TRANSMITTAL.

OTTAWA, February 27, 1907.

To the Honourable
The Minister of Agriculture.

SIR,—I have the honour to submit for your approval Bulletin No. 14, Dairy and Cold Storage Commissioner's Series, entitled 'Apparatus for the Determination of Fat and Water in Butter,' by Mr. F. T. Shutt, M.A., Chemist, Experimental Farms, in which Mr. Shutt gives the results of investigations made at my request for the purpose of ascertaining the reliability and practicability of certain apparatus, now being offered for sale, for the purpose of determining the percentage of water and also the percentage of fat in butter. Owing to the provisions of the Butter Act, 1903, there is a demand on the part of buttermakers, and butter dealers, for a simple and accurate method of determining the percentage of water in butter. I consider the information herein contained of sufficient value and interest to warrant a special publication. I beg to recommend, therefore, that it be printed for general distribution.

I have the honour to be, sir,
Your obedient servant,

J. A. RUDDICK,
Dairy and Cold Storage Commissioner.

APPARATUS FOR THE DETERMINATION OF FAT AND WATER IN BUTTER.

By Frank T. Shutt, M.A.,
Chemist, Dominion Experimental Farms.

In 1904 the writer examined and reported upon the 'Carroll tester' and 'Geldard butter tester,' both being forms of apparatus devised in England for the ready estimation of water in butter. The former was not found trustworthy, and could not be recommended even where approximate results only were required. The latter gave extremely satisfactory results, the data being in close accord with those obtained by analysis, provided the operation was carried out with care.*

During the past season two further pieces of apparatus have been examined—the one for the determination of fat, and the other of water, in butter. Both are from the manufactory of the Wagner Glass Works, New York, U.S.A.

THE WAGNER BUTTER TEST BOTTLE.

By means of this bottle, it is stated, a practical and convenient method is offered for obtaining the percentage of fat in any sample of butter. The directions furnished with the bottle give no definite information regarding the quantity of acid to be used, nor the temperature at which the fat column is to be read.† We found at the outset of this investigation to ascertain its accuracy, that these were matters of very considerable importance, and that the percentages of fat as indicated in the graduated tube varied within quite wide limits, according to the quantity of acid employed and, more particularly, with the temperature of reading.

On writing the Wagner Glass Works, New York, on these points, they replied as follows:—

1. About 2 cc. of normal strength sulphuric acid are required for making the test in the Wagner Test Bottle.

2. The fat column should be read at about 140° F. The easiest way to control temperature is to use a water-bath at 140° F. and leave the bottle in it for about [illegible] utes. The contents of the bottle will take the same temperature as the water-bat

*Chemical investigations relating to dairying undertaken in 1904, Bulletin No. 6, Dairy Commissioner's Branch, Ottawa.

†Directions accompanying the Wagner Butter Test Bottle: 'After bottle being balanced on a scale add 9 grams of butter, solid form, in the side tube (funnel-shaped tube) the bottle is then to be placed in hot water, which will soon melt the butter and the butter runs into the bottle. A small amount of acid should then be added and the bottle is then to be placed in a tester, the test is completed as any cream test. To read off the butter-fat, fill the side tube (funnel-shaped tube) with hot water which will raise the fat column in the graduated neck. by gently pressing down or gently drawing up the rubber cork on top of the graduated neck, the fat column may be moved in the graduated neck so as to bring the lower end of the fat column level with the zero mark which is indicated by a ring below the bulb, and the percentage of fat may be read directly, without the use of dividers, or other measuring tools.'

Quantity of Acid.—As illustrative of our results, using varying amounts of acid, the following data may be given: the strength of the sulphuric acid was 1·82-1·83 Sp. Gr.

Using 2 cc. acid: No clear or accurate reading could be obtained owing to foaming and partial charring of the fat. In every case the reading was too high, that is, greater than indicated by gravimetric analysis. The excess sometimes was as much as 2 per cent.

Using 1 cc. acid: With this quantity no difficulty was experienced in obtaining clear readings. If the acid is added slowly to the melted butter in the bottle, shaking meanwhile, there will be no foaming or charring of the fat. Subsequent experiments proved that 1 cc. acid was sufficient to completely separate all the fat in the butter.

Temperature of Reading.—The reliability of the results from this 'test bottle' depends largely upon the temperature of the contents of the bottle when the fat column is read. This will be apparent from the subjoined data:—

Fat in butter, by gravimetric analysis, 84·02 per cent.

	Fat in Wagner's Bottle, using 1 cc. acid. Per Cent.
*Trial A—	
When taken out of steam Babcock machine about 195° F. . . .	87
**After standing in water for 10 minutes at 122° F.	84
*Trial B—	
When taken out of Babcock machine about 195° F.	87
After standing in water at 122° F. for 10 minutes.	84
Left until temperature of water was 90° F.	82·4
Trial C—	
When taken out of Babcock tester about 195° F.	87
After standing 5 minutes in water and read at 140° F. . .	84·02
After standing 5 minutes in water and read at 135° F. . .	84·15
After standing 5 minutes in water and read at 122° F. . .	84·00

Trials were then made with other samples of butter, as follows:—

Trial D—	By Wagner. Per Cent.	By Gravimetric Analysis. Per Cent.
When taken out of Babcock.	87·8	84·67
After standing in water 10 minutes at 122° F.	84·7	
Trial E—		
When taken out of Babcock.	88·0	85·09
After standing in water 10 minutes at 122° F.	85·0	

Though several butters were tested by this method, the results are from one bottle only. Two had been obtained for test, but unfortunately one of them had broken in the Babcock tester at the beginning of this investigation. The bottle tested gave re-

*Trials A. and B. were made previous to reception of the letter of the Wagner Glass Works already referred to. The original printed instructions make no mention of any particular temperature at which to read the fat column.

**The bottle on being taken out of the machine was put in a vessel of water holding about three pints, at the temperature indicated, viz.: 122° F. At the end of 10 minutes the temperature of this water had fallen from 1° to 2° F.

sults consonant with those obtained by gravimetric analysis, by using 1 cc. acid and reading the fat column after placing the bottle (direct from the machine) for 10 minutes in water that had a temperature of 122° F.

The following notes regarding the working of the test, when using a steam Babcock tester, will be of service:—

After weighing the butter in the side tube, the bottle is placed in the tester and revolved in the heated machine for about 2 minutes. The acid is now added drop by drop, with constant shaking of the contents of the bottle. The bottle is then replaced in the tester and revolved for 5 minutes. Hot water is then added to bring the column of fat into the graduated neck, and the bottle again revolved for 2 minutes. It is then taken out of the tester and placed in a vessel of water at 122° F. for 10 minutes, and read.

The foregoing work was done in July. In October, further directions for using the test bottle were received from the Wagner Glass Works, as follows:—

'After bottle being balanced on a scale add 9 grams of butter, solid form, in the side tube (funnel-shaped tube). The bottle is then to be placed in hot water which will soon melt the butter and the butter runs into the bottle. Rinse down the small amount of butter fat adhering to the wall of the funnel with 8·8 cc. hot water, and add 8·8 cc. of acid, mix well and place the bottle in a tester; whirl for about 5 minutes.'

The trial was made, using these instructions, with a butter showing 84·09 per cent fat by gravimetric analysis. The reading was good, the line of demarkation being clear and sharply defined. The fat gave no indication of charring.

	Fat. Per Cent.
When taken out of machine, temperature about 186° F., the reading was	88·0
Allowed to stand 10 minutes in water at 140° F. the reading was	85·8
Allowed to stand in water until 122° F., the reading was	85·1

As without a special pipette or burette the quantity 8·8 cc. would be very difficult to measure, a trial was made using 5 cc. of water and 5 cc. of acid—a 5 cc. pipette being easily obtained. This trial gave results equally good with the foregoing, as the following readings show:—

	Fat. Per Cent.
When taken out of the machine, temperature about 186° F., the reading was	88·0
Allowed to stand 10 minutes in water at 140° F., the reading was	85·2
Allowed to stand in water until 122° F., the reading was	85·0

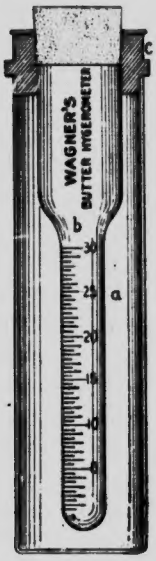
The line of demarkation was clear and the fat a good colour.

From the foregoing it is evident that good readings may be obtained either by using 1 cc. acid; or by employing 5 cc. or 8·8 cc. acid when an equal volume of hot water is first added to the melted butter.

It will be observed that the temperature at which the reading is made is a matter of great importance and we advise in this connection placing the test bottle as taken out of the machine in a vessel containing from 2 to 3 pints of water at 122° F. The water is maintained at this temperature for at least 10 miinutes in order to allow the contents of the test bottle to reach this temperature, and the reading then made. The

Babcock machine used in this investigation was operated directly by steam, the escaping steam raising the temperature of the tests as already indicated.

THE WAGNER BUTTER HYGROMETER.



This simple piece of apparatus has been devised for ascertaining the percentage of water in butter. The following description and directions are from the printed circular accompanying the bottles:—

The illustration represents a cross section of the instrument. A bath 'a' is filled with water held at 140° F., the lower half of the test tube 'b' bears a scale graduated from 0·30 per cent, from which the moisture content of the sample can be read directly. The tube is closed by soft rubber stopper which fits snugly into the top. The support 'c' prevents the graduated test tube from falling into the water bath.

In making the test, 18 grams of butter are weighed into the graduated test tube, the tube being closed by the soft rubber stopper and then inserted in the water bath cylinder at about 140° F., the graduated test tube being held in position by a soft rubber support. As soon as the butter has melted completely the apparatus is placed in a Babcock testing machine and whirled for about 10 minutes, if hand Babcock testing machine is used, the water bath should be reheated two or more times during whirling, the water content of the butter will soon collect at the bottom of the graduated test tube and can be read directly from the scale, by salted butter 2 per cent should be allowed for salt.

Four hygrometers were tested, and the first trial resulted in the following data:—

Hygrometer—As taken out of machine (steam turbine)—

No. 1	15	p.c. water
No. 2	15	"
No. 3	15·5	"
No. 4	14·5	"

After standing the cylinders for 10 minutes in water at 140° F., the readings were uniformly 0·5 per cent lower than the above.

The percentage of water as found by gravimetric analysis, 13·13.

If from these readings 2 per cent for salt is deducted, according to printed instructions, we obtain:—

	Per Cent.
No. 1	13·0
No. 2	13·0
No. 3	13·5
No. 4	12·5

Owing to the curd not settling properly, the readings were very difficult to make. In no case was there a sharp line of demarkation between the water and the fat. Further, the line of division between the mixed water and curd and the fat when the bottle was taken out of the machine was at an acute angle in the graduated portion of the tester. No subsequent treatment was found effective in obtaining any horizontal line of demarkation. These facts made the readings at best but approximate.

On September 6, and subsequent to the foregoing work, new instructions were received from the Wagner Glass Works regarding the use of this apparatus, as follows:—

Directions for Reading the Wagner Butter Hygrometer to Correspond with Chemical Analysis.

'It will be observed that there is a sharp layer of water as well as a sharp layer of caseine (the caseine is combined with water). Every 1 per cent combined water and caseine indicated on the hygrometer should be read as 0·1 per cent caseine. We have come to this result by removing the caseine of the combined caseine and water by drying same. For instance, if the butter hygrometer shows:

	Per Cent.
A sharp water line of	6
A sharp combined caseine and water line of	11
The moisture would be	15·9
Caseine if dried up to powder	1·1
Actual butter-fat	83

'If it is not desired to determine the amount of caseine in butter, the clear water content as well as the combined caseine and water may all be read as moisture. If the hygrometer shows 14 per cent moisture the butter-fat will be 86 per cent.'

Further trials were then made, two butters being selected, the one with a high and other with an average, water content.

Readings from butter A., showing 18·17 per cent water by gravimetric analysis:—

No. 1. Reading of water line 15
Reading of combined water and curd line 21
21 — ·6 = 20·4 — 2 per cent for salt = 18·4

No. 2. Reading of water line 16
Reading of combined water and curd line . . . . . 20·5
20·5 — ·45 = 20·05 — 2 per cent for salt = 18·05

No. 3. Reading of water line 14
Reading of combined water and curd line 21
21 — ·7 = 20·3 — 2 per cent for salt = 18·3

No. 4. Reading could not be made owing to uncertainty regarding the line of demarkation between fat and water. The readings were made as the cylinders were removed from the machine. Difficulty was again encountered in reading owing to the slanting line of demarkation.

Duplicates proved unsatisfactory, as will be gathered from the following results:—

No. 1. Reading of water line 13
Reading of combined water and curd line 17
17 — ·4 = 16·6 — 2 per cent for salt = 14·6

No. 2. Reading of water line 13
Reading of combined water and curd line 20
20 — ·7 = 19·3 — 2 per cent for salt = 17·3

No. 3. Reading of water line 15
Reading of combined water and curd line 20
20 — ·5 = 19·5 — 2 per cent for salt = 17·5

No. 4. Reading impossible, owing to imperfect separation into layers.

In only a small percentage of the above trials could the readings be made with certainty. In the majority of instances the demarkation between the fat and water and curd was so confused that an approximation only could be arrived at.

After the cylinders had been used several times it was found that the soft rubber stoppers were very apt to come out during the whirling in the Babcock, with the result that the determination was lost.

Readings from butter B., showing 13·06 per cent water by gravimetric analysis:—

Tester No. 1. No line of demarkation between water and mixed water and curd, the reading of the column of the aqueous mixture being 18·5 per cent.

NOTE.—If this is considered as all water (see above instructions), and 2 per cent deducted for salt, the corrected reading for water would be 16·5 per cent.

Tester No. 2. Reading of water line.. 6
Reading of combined curd and water line 16
16 — 1·0 = 15 — 2 per cent for salt = 13 per cent.

Tester No. 3. Reading of water line.. 6
Reading of combined curd and water line.. .. 16
16 — 1·0 = 15 — 2 per cent for salt = 13 per cent.

Tester No. 4. Reading of water line.. 6
Reading of combined curd and water line.. .. 16·5
16·5 — 1·05 = 15·45 — 2 per cent for salt = 13·45 per cent.

Several of these readings were mere approximations owing to indistinctness or lack of sharpness between the several layers.

The writer, after considerable experience with this hygrometer, cannot speak in unqualified terms as to its general satisfactoriness. It is quite true that in a number of trials the readings, after calculations, gave data sufficiently near the true water content for all practical purposes, but the uncertainty in obtaining distinct layers which can be readily read off seems to be too great to make the instrument of value in the warehouse or dairy, where it is particularly desirable that the readings should not only be fairly accurate, but also easily and quickly made.

The writer desires to record his thanks to Mr. A. T. Charron, Assistant Chemist, for much valuable help in these investigations.

LIST OF PUBLICATIONS

OF THE

DAIRY AND COLD STORAGE COMMISSIONER'S BRANCH

Date Issued.	No.	Title.
1905	1	List of some British Importers of Farm Products.
1905	*2	Milk for Cheese Factories.
1905	*3	Milk for Creameries.
1905	4	Some Phases of Dairying in Denmark.
1905	5	Improvement of Dairy Herds.
1905	6	Chemical Investigations Relating to Dairying in 1904.
1905	7	List of Exporters of Some Canadian Products.
1906	8	Some of the Factors that Control the Water Content of Butter.
1906	9	Instructions for Testing Individual Cows, &c.
1906	10	Creamery Cold Storage.
1906	11	General Instructions *re* Fruit Marks Act as Amended, 1902 and 1906.
1906	12	Cow Testing Associations, with Notes on the Sampling and Testing of Milk.
1907	13	Sweet-Cream Butter.
1903		Notes for Cheesemakers.
1905		Report of the Dairy Division, 1904.
1905		Report of Extension of Markets Division, 1904.
1905		Evidence of J. A. Ruddick, before Committee on Agriculture and Colonization, 1905.
1905		Evidence of A. McNeill, before Committee on Agriculture and Colonization, 1905.
1906		Evidence of J. A. Ruddick, before Committee on Agriculture and Colonization, 1906.
1906		Proceedings of the Second Conference of Fruit Growers of the Dominion of Canada.
1906		Report of the Dairy Commissioner, January, 1905, to March, 1906.

Any of these publications will be sent free of charge on application to the Dairy and Cold Storage Commissioner, Ottawa, Ont.

*A sufficient number of bulletins No. 2 and 3, will be sent to the manager of any cheese factory or creamery, to supply one to each patron.